DATA VALIDATION SOP

ORGANOTINS

STANDARD OPERATING PROCEDURE
DATA VALIDATION OF ORGANOTIN ANALYSIS

A. INTRODUCTION

This Standard Operating Procedure (SOP) provides guidance for the validation of organotin analytical data generated from the analysis of water, soils, sediments, or tissue samples. The validation procedure was based upon the EPA Region II data validation SOP for Pesticides/PCBs (EPA 1992)[1]. A copy of the analytical protocol is provided as an appendix to the QAPP.

B. DATA VALIDATION PROCEDURES

1. Holding Times

Criteria: Holding times are established for both extraction and sample analysis. For aqueous, soil and tissue matrices, the extraction must begin within 7 days from sample collection, with analysis within 40 days of extraction.

Actions: The actions are summarized in Table 1. Actions may be taken for either time from sampling to extraction or time from extraction to analysis, but not both. If neither criteria are met, the exceedance which occurs first (i.e., time to extraction, or the criteria which results in additional qualifications or the "R" qualification takes precedence.

Table 1. Summary of Actions for Holding Times

Time from Sampling to Extraction			
Matrix	0-7 days	7-14 days	> 14 days
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects
Time from Extraction to Analysis			
Matrix	0-40 days	40-80 days	> 80 days
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects

2. Initial Calibration

The gas chromotograph undergoes a 5 point initial calibration with all of the targeted analytes. The laboratory may use either a linear regression, or calculate response factors, to ensure proper linear response of the instrument.

Criteria: If linear regression is performed, the laboratory must demonstrate that the regression coefficient ("r") is greater than or equal to 0.995.

If calibration factors are used, the laboratory must demonstrate that the relative standard deviation of the calibration factors is less than 30%.

Actions: The actions are summarized in Table 2.

Table 2. Summary of Actions for Initial Calibration

Initial Calibration using Regression			
Matrix	R ≥ 0.995	0.95 < r < 0.995	R < 0.95
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects
Initial Calibration using Calibration Factors			
Matrix	RSD < 30%	30% < RSD < 80%	RSD > 80%
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects

3. Continuing Calibration

The gas chromatograph undergoes a 1 point daily calibration with all of the targeted analytes to ensure consistency with the initial calibration.

Criteria: If linear regression was used for the initial calibration, then the continuing calibration result calculated from the linear regression must agree within 25% of the nominal concentration.

If calibration factors were used for the initial calibration, the laboratory must demonstrate that the percent difference between the mean calibration factors from the continuing calibration and the average response factor from the initial calibration is less than 30%.

Actions: The actions are summarized in Table 3.

Table 3. Summary of Actions for Continuing Calibration

Calibration using Regression			
Matrix	75 – 125% of nominal value	20 – 74% or 126 – 180% of nominal value	< 20% or > 180% of nominal value
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects
Calibration using Calibration Factors			
Matrix	%D < 30%	30% < %D < 80%	%D > 80%
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects

4. Surrogate Recoveries

For soil, water and tissue samples, surrogate is added to the sample extract prior to derivitazation.

Criteria: The laboratory must demonstrate that the surrogate recovery falls between 30 and 160%. If the initial analysis fails this criterion, then the samples must be reprocessed once.

Actions: The actions are summarized in Table 4. Chromatograms should be reviewed prior to taking any action to ensure that there were not interferences or coeluting peaks which resulted in the surrogate recoveries falling outside of the control limit. If it is the Reviewer's professional opinion that the non-compliant surrogate recoveries were due to interferences or co-eluting peaks, no action may be required.

No action is required if the surrogates were diluted out.

Table 4. Summary of Actions for Surrogate Recoveries

	%R < 10%	10 < %R < 30%	30 < %R < 160%	%R > 160%
Positives	"R"	"J"	Accept	"J"
Non-Detects	"R"	"UJ"	Accept	Accept
Note: No action is taken if the surrogates are diluted out.				

5. Method and Field QC Blanks

Method blanks are prepared by the laboratories to identify the potential for laboratory contamination. Field QC blanks are provided by the field team to identify potential contamination by the targeted analytes which may be introduced during field activities.

Criteria: There should be no detected targeted analytes in the method or field QC blanks.

Actions: If the targeted analytes are reported in the method or field QC blanks, and are present at a concentration less than 5 times the blank concentrations in the samples (adjusting for matrix variation and sample size), the reported sample results are potential false-positives and should be qualified with a "U". No action is taken if the sample concentration is greater than 5 times the method or field QC blank results. Likewise, when associated samples are "not detected" for the analyte, no qualification of data is necessary.

6. Data Calculations and Reported Concentrations

The reported concentrations for the targeted analytes in at least one sample in the Sample Delivery Group should be recalculated and verified. If a discrepancy is noted, the laboratory should be contacted to provide a sample calculation. Corrective actions may be required, as appropriate.

If the laboratory employs a confirmation column to verify sample identifications, it is recommended that the Reviewer verify that the concentrations on both columns are comparable. If significant differences are noted which are attributable to interferences or coeluting peaks, the Reviewer should consider reporting the results from the confirmation column.

7. Sample Moisture Contents

If the sediment samples exhibit moisture contents in excess of 50%, the actions in Table 6 should be taken.

Table 6. Actions for Excessive Moisture Contents in Sediments

	%Moist < 50%	50 < %Moist < 90%	%Moist > 90%
Positives	Accept	"J"	"M"
Non-Detects	Accept	"UJ"	"M"

8. Project Specific Requirements

If the direction of the bias can be determined from the analytical results, then the data reviewer can apply the following data qualifiers:

- Potential high bias: JH or UJ
- Potential low bias: JL or UJL

If the direction of the bias can not be determined, or the assessment of bias is contradicted by the quality control results (e.g., holding time exceedance with high surrogate recoveries), then the overall bias is undetermined, and the "J" or "UJ" qualifiers are appropriate.